STUDIES ON THE PREPARATION AND PROPERTIES OF IRON/STEEL STRIPS MADE FROM PREFORMED IRON ORE SUPER-CONCENTRATE BLOCKS

by

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C E R T I F I C A T E

This is to certify that the present work 'STUDIES ON THE PREPARATION AND PROPERTIES OF IRON / STEEL STRIPS MADE FROM PREFORMED IRON ORE SUPERCONCENTRATE BLOCKS ' has been carried out by Mr. Subodh Kumar under my supervison and it has not been submitted elsewhere for a degree.

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A C K N O W L E D G E M E N T

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SUBODH KUMAR

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SYNOPSIS

One of the various possible variants of 'Direct Strip Process' consists of compacting a block from a mixture of iron oxide superconcentrate powder and a binder. The 'green' superconcentrate block is simultaneously reduced and sintered at an elevated temperatures using a solid reductant such as coal or charcoal. The resulting sponge iron block is subsequently hot rolled to form a fully densified strip, which is then cold rolled and annealed to produce the finished strip.

The present study deals with finding out the suitability of saw dust charcoal as a reductant for the block variant of direct strip process. Simultaneous carburization which may occur during reduction of superconcentrate block in the packed bed of saw dust charcoal, and decarburization which may occur during preheating of the reduced block in hydrogen atmosphere prior to hot rolling, has also been studied. Finally, the amount of cold rolling has been optimized to obtain the best mechanical properties of the cold rolled and annealed finished strip.

It has been found that saw dust charcoal is suitable to be used as a reductant for the 'block variant' of direct strip process. It has also been found that the simultaneous carburization does take place during reduction of the block; the carbon content of the reduced block increases with the reduction time and it varies from corner to the centre of the block. However,

decarburization takes place during preheating in hydrogen atmosphere prior to hot rolling, and carbon content is reduced to a very low level in the hot rolled strips. The best mechanical properties have been found for the finished cold rolled and annealed direct strips which have been given 50-60% reduction in thickness during cold rolling.

CHAPTER 1

INTRODUCTION

A large fraction of total " production " is rolled in the form of strips, for most common metals. In case of steel, the flat steel products contribute around 20% of the total production, on a world wide scale and this percentage is rising. Most thin strips (thickness $\sim$ 0.25 to 0.1 mm) are produced from large size ingots of thickness $\sim$ 300 mm . Thus the amount of deformation required to produce the finished strip is enormous. It causes a major increase in capital equipment and operating costs, and the yield of finished metal strips or sheets is low, of the order of 70% for steel. Because of extensive hot and cold rolling, the energy requirements are also high.

The above drawbacks can be minimised by starting with a thinner slab. In continuous casting, the thickness of the continuous cast slab, which is the starting material for the production of thin strips, is 100 - 150 mm for steel. This reduces the hot rolling to a considerable extent. Although, the continuous casting has several advantages over static casting, it's introduction does not radically change the situation for steel. The thickness of the continuous cast slab can not yet be further reduced because of the engineering problems associated with it, although efforts are being made worldwide to cast thin strip continuously which can be hot and cold rolled to produce the finished strip.

Further, the conventional method of steel strip production, to be economically viable, requires large plant size, the capacity being not less than 1-3 million tonnes per annum, depending on the location. The large units are capital intensive and have a long gestation period. The experience of mini-mills in U.S.A. shows that it is much more economical to run smaller plants at high utilization factor than larger plants at a lower utilization factor.[1]

From the foregoing discussion, it is clear that the conventional thin steel strip manufacturing industry is highly capital intensive and there is much room for the development of the alternative routes of thin strip making from an economically viable small or medium size plant requiring low capital investment.

1.1 Powder Metallurgy Routes for Making Thin Steel Strip.

Thin strip making routes based on powder metallurgy are most important amongst the alternative routes developed to date. The economically viable minimum plant capacity for making steel strip by powder metallurgy route is 50,000 t to 100,000 tons per year. The capital cost of P/M plant is approximately 30% less than the conventional plants.[2] The product yield for the P/M plant is about 90% . The reduction to produce the final finished strip is of the order of 5 : 1 . The energy requirements for the production of the steel strip by the P/M route is 27 GJ_t/ t to 33 GJ_t/ t of the finished strip as against 34.6 GJ_t/ t for the conventional route[3].

The starting material for making steel strip by P/M route is iron/steel powder. This powder is compacted into a ' green" strip which is a mechanically bonded metal powder formed into a strip which is relatively porous and brittle. This can be done by the method of roll compaction of the loose powder or by first making a coherent and flexible strip from a slurry made from powder and suitable binder and then roll compacting it. The green strip thus obtained is then sintered at a sufficiently high temperature (around 1100°C to 1150°C for iron/steel) and hot rolled in a single pass to produce fully dense strip, which is subsequently cold rolled and annealed to make finished strip. Alternatively, the sintered strip may be densified by repeated cold rolling and annealing cycle to make fully dense finished strip.

1.2 Integrated Powder Technology Routes for Making Thin Steel Strips

The P/M route, as outlined above, however, suffers from economic constraints, as at present the cost of iron/steel powder is more than the cost of steel slab. It is partly due to low demand for iron/steel powder at present. It is therefore necessary to integrate this separate step of powder production into the process of strip making itself to make its production economically viable. It will also reduce the number of unit operations and will make the process economically more attractive by lowering the capital equipment and operating costs.

A couple of such methods have been developed, and one such method is " Direct Strip Process '.

Direct Strip Process consists of making a homogenous and free-flowing slurry from iron oxide superconcentrate powder (iron oxide ~ 99%) and a water soluble binder[1-3]. The slurry is cast into a strip form and dried. Subsequently it is roll compacted to increase its strength. The roll compacted super-concentrate " green' strip - then simultaneously reduced and sintered at a high temperature to form a sponge iron strip which is subsequently hot rolled to produce fully dense strip. It is pickled, cold rolled and annealed to produce the final finished strip. It is apparent that the above route does not involve the production of iron/steel powder at any intermediate stage, and yet operates on the basic principles as that of the traditional P/M route described earlier. The starting material in this process is iron ore superconcentrate and the cost of beneficiation to get the ore of such high purity is not much, keeping in view the fact that no further refining is needed at any intermediate stage. L.K. .B. of Sweden has demonstrated that the cost of magnetite superconcentrate is not more than 50% of crude ore price per unit of iron[9]. The various unit steps of steel strip production from iron ore superconcentrate are also integrated in the sense that the various stages of the complete process follow one another immediately, and the product of one stage is not subjected

to handling or removal to storage before proceeding to the next. For these reasons, such a route has been termed as integrated power technology route. The direct strip process is based on gaseous reductants such as pure H_2 or a mixture of H_2 and CO gas derived from the steam reforming of naptha cuts.

1.3 A New Route for Making Thin Strips Directly from Iron Ore Superconcentrate

The direct strip process as outlined above does not permit the use of solid reductants such as coal, charcoal etc. One such variant of direct strip process has been described[10] which allows the use of coal as a reductant. This route has been shown in Fig. 1.1. It consists of mixing iron ore superconcentrate powder (iron oxide ~ 99%) with a water soluble binder such as Acacia arabica, in such a proportion that each particle of powder is just coated with the binder solution. This mixture is compacted in a die to a block shape which is subsequently reduced by coal surrounding the block kept inside a muffle at about 1150°C. The reduced sponge iron block is then hot rolled to a fully densified strip which is further cold rolled and annealed to produce the finished strip.

The above mentioned route has been successfully studied by Vethanayagam[10]. Magnetite superconcentrate powder (iron oxide ~ 99.5%) supplied from L.K.A.B., Sweden was used as the starting raw material;

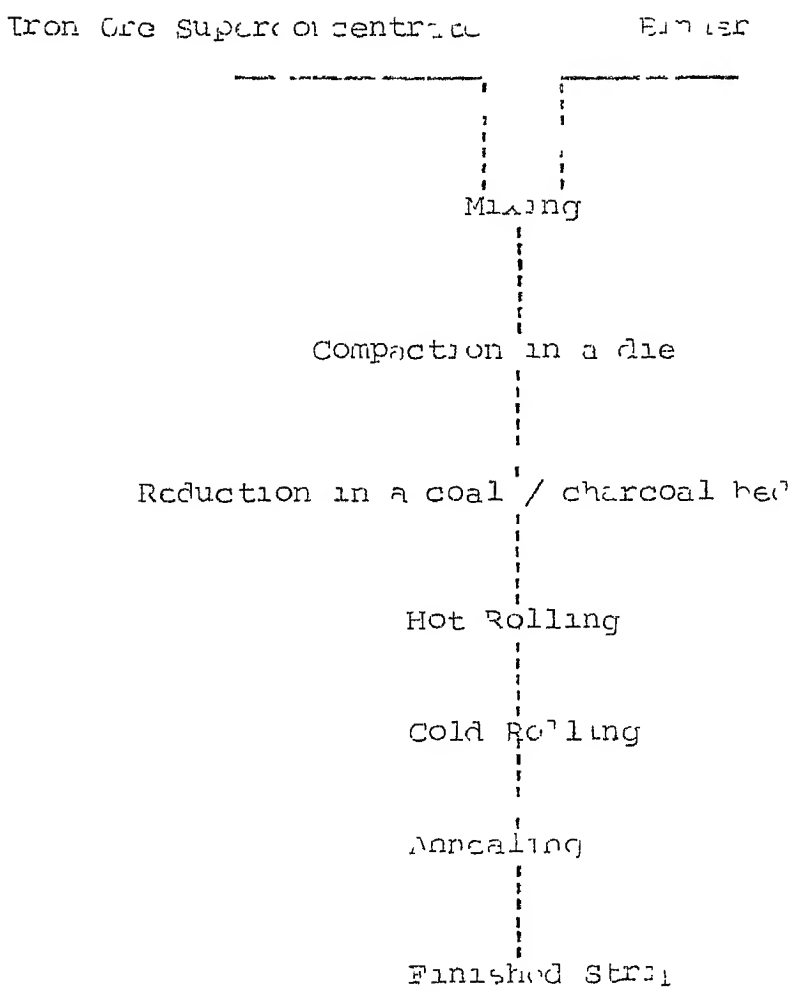

Fig. 1.1 Block variant route of direct strip process for making thin steel strips directly from iron ore superconcentrate

Acacia Arabica was used as a binder ; and three varities of coal- washed and middling grade coal, devolatalized brown coal and conventional charcoal were tried as reductants. There were, however, few problems encountered with these solid reductants. The washed and middling grade coal which contained a large amount of volatile matters created serious practical problems because of the sticking of the entire coal mass to the inner wall of the metallic tube. Hence it was not considered suitable for further investigation. Conventional charcoal and devolatalized brown coal did not pose any such problem but another problem was encountered which was related with the size and shape of the coal particles. Use of coarse size coal, say, > 10 mm, resulted in the reoxidation of the freshly reduced sponge iron block during cooling inside the furnace, due to passage of atmospheric air to the block through permeable coal bed. Use of -6 (3.3 mm) mesh size coal prevented the passage of air through coal bed, but use of fine size of these types of reductants left behind a few Klinkers on the surface of the block and some times the coal particles were embeded on the surface. These Klinkers or the embeled coal particles could be removed from the surface but only at cost of addition of one extra unit step of machining of the surface of the reduced sponge iron block.

1.4 The Requirements for Solid Reductant

To be used as a reductant for ' block' variant of direct

strip process, the coal/charcoal should satisfy the following requirements -

(1) It should have a good reduction behaviour, i.e., it should not take a very long time to fully reduce the iron ore superconcentrate block to sponge iron. It means that -

(a) activity of carbon in coal particles should be high, and

(b) the size and shape of the coal particles should be such that they make an intimate contact with the surface of the block.

(2) The coal mass should not stick to the walls of the furnace or the surface of the block.

(3) The coal size should be fine enough not to cause the problem of reoxidation of the reduced block during cooling inside the furnance.

(4) The shape and surface properties of coal particles should be such that they do not deteriorate the surface of the block.

(5) It should be cheap and readily available.

None of the reductants, tried earlier, satisfy all of the above requirements together, so it remains to find out a suitable solid reductant for the block variant of direct strip process and present investigation makes an attempt in this endeavour.

The mechanical properties of the finished strip obtained by direct strip process[11] (referred to as ' direct strip ', hence onwards) were found to be comparable with those of low carbon steel strip. But the process of carburization of the sponge iron block is expected to take place during it's reduction in the packed bed of coal/charcoal, in which it is kept at 1150°c for 3 hours, and the reduced block is expected to contain a very high percentage of carbon, which is surprisingly not reflected in the mechanical properties of the resultant strip. Therefore, it was imperative to investigate whether the process of carburization was really taking place during the reduction of the block, and if yes, then by what mechanism the carbon level could go down drastically in the finished strip to give mechanical properties comparable is low carbon steel. Such a study was not made by Vethanayagam[10], and has been undertaken in the present investigation.

Finally, the mechanical properties of the finished cold rolled strip/sheet depend on the amount of cold rolling deformation given prior to final annealing. Vethanayagam[11] has found different mechanical properties of cold rolled and annealed direct strip for different amount of reduction in thickness of strip during cold rolling. But no attempt was made to optimize the percentage reduction of thickness of strip during cold rolling to obtain the best mechanical properties. The present investigation is also concerned with the optimization of cold rolling deformation prior to final annealing.

CHAPTER 2

OBJECT OF THE PRESENT INVESTIGATION

The aims of the present investigation are as follows -

(1) To study the suitability of saw dust charcoal as a reductant for magnetite superconcentrate compacts.

(2) To study the simultaneous carburization which is occuring during the reduction of magnetite superconcentrate compacts in a packed bed of saw dust charcoal and the decarburization of the reduced compacts prior to hot rolling .

(3) To produce fully dense hot rolled and cold rolled strip from the sponge iron blocks and to optimize the amount of cold rolling to obtain the best mechanical properties of the finished strips.

CHAPTER 3

RAW MATERIALS AND EXPERIMENTAL PROCEDURES

3.1 Raw Materials

3.1.1 Magnetite Superconcentrate

Magnetite superconcentrate supplied by L.K.A.B. of Sweden was used. The chemical composition and sieve analysis are shown in Tables 3.1 (a & b). For all the experimental purpose powder of size -300 mesh was used.

3.1.2 Acacia Arabica

Acacia Arabica was procured from the local market. The chemical composition of this binder was not available from the local supplier. The density of Acacia Arabica determined by Archimedes' principle was 1.44 gm/cc and residue content of small grannules was found to be 2.68 wt %, while residue content of big grannules was found to be 1.33 wt %.

3.1.3 Saw Dust Charcoal

The saw dust charcoal was obtained by heating saw dust at 800^{o}C in a muffle in the absence of air for 3-4 hours. Fumes in very large quantities were released during this process. This coal was equisized and rice-grain shaped.

3.1.4 Gases .

IOLAR 3 Grade hydrogen and standard nitrogen supplied from cylinders were used. The composition of IOLAR 3 Grade hydrogen gas is given in Table 3.2.

TABLE 3.1a CHEMICAL COMPOSITION OF MAGNETITE SUPERCONCENTRATE

Constituents	Weight %
Fe	71.7 - 71.8
Fe^{++}	23.8
P	0.001
S	0.003
CaO	0.05-0.07
MgO	0.15-0.20
Al_2O_3	0.15-0.20
SiO_2	0.05-0.10

TABLE 3.1b SIEVE ANALYSIS OF MAGNETITE SUPERCONCENTRATE

Size	%
-417 /um	100
-208 /um	99.9
-104 /um	96.3
- 62 /um	85.3

Table 3.2 Composition of IOLAR 3 Grade hydrogen gas

Components	ppm
O_2	6
H_2O	6
CO_2	1.0
CO	1.0
Oxides of N_2	1.0
N_2	2.50
Hydrocarbon	2.5
Sulphur compounds	0.1
H_2	rest

3.2 Experimental Procedure

3.2.1 Preparation of Green compacts

First a binder solution was prepared. The amount of water for preparing the solution was calculated on the basis of the fact that the ore to water ratio was 20 by weight [10]. 1 Wt.% (of the ore) binder, i.e., Acacia Arabica was added to the calculated amount of water and the solution was heated at around 80°c for fifteen minutes to get the complete dissolution.

The binder solution was added to the already weighed amount of superconcentrate iron ore powder in a beaker and it was thoroughly mixed with the help of motor driven stirrer. Immediately after mixing, the powder was compacted in a rectangular die by applying a pressure of 45 MN/m^2 into the blocks of size 71 mm x 48 mm x 6 mm having a density of 65% of the theoretical density and having a sufficient strength for further handling of the block. These blocks were dried in an air oven at a temperature of 100-125°c for a duration of 3-4 hours.

3.2.2 Combined Reduction and Sintering of Green Compacts

Fig. 3.1 shows a view of typical superconcentrate green compact which was reduced and simultaneously sintered as well. A specially designed furnance as shown in Fig. 3.2 was used for this purpose. It consisted of a vertical nimonic tube of internal diameter of 100 mm, with both ends

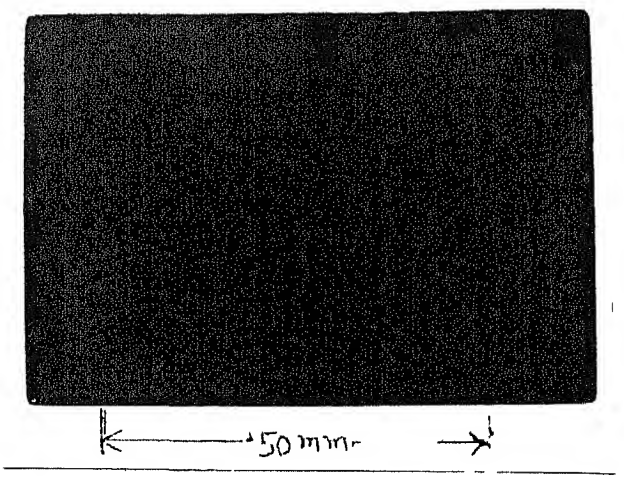

Fig.3.1. A View of Superconcentrate Green Compact.

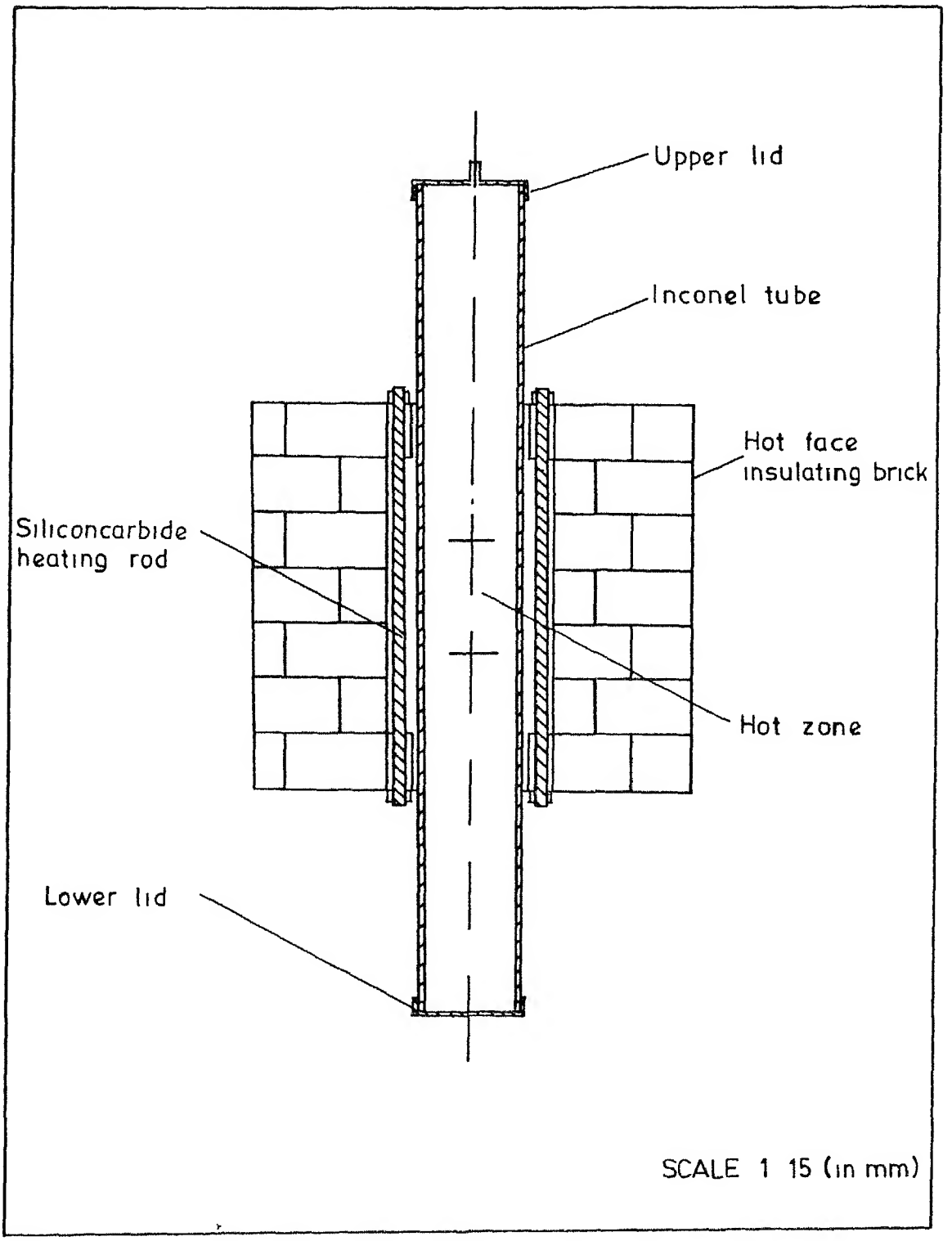

Fig. 3.2 A cross sectional view of the reduction furnace.

open. This type of furnace facilitates easy loading and unloading of the charge material. The tube was externally heated with silicon carbide heating rods. The furnace had a constant hot zone of about 120 mm situated at the centre.

The lower end of the tube was closed with a lid and saw dust charcoal was charged upto middle and was rammed. Green oxide superconcentrate compact was placed on it, and more saw dust charcoal was charged upto the upper end of the tube then the upper end was also closed with a lid.

The charged tube was heated to 1150°c. The temperature was attained in about 45 minutes. The charge was kept at this temperature for 3 hours and then the furnace was switched off. It took about 8-10 hours for the charge to cool down to room temperature. Subsequently the lower lid was removed and the entire charge was withdrawn from the tube. The sponge iron block was removed from the charcoal which was reused for the next charge.

3.2.3 Hot Rolling of Sponge Iron Block

The sponge iron block obtained after complete reduction of magnetite superconcentrate compacts contained about 70% - 80% porosity, which correspond to density range of 1.7-2.5 g/cc. In order to obtain a fully dense strip it was necessary to densify the block by hot rolling. The preheating and hot rolling of such a material must be done in a protective atmosphere to avoid oxidation caused by interconnected pores. The preheating of the sponge iron block was done

in hydrogen atmosphere. One end of the preheating chamber was closed while the other end had an extended zone projection outside the furnace.

The preheating chamber, which was a nimonic tube of 100 mm internal diameter, contained a flat plate as a base for the sponge iron block. The reheating was done at 1150°C for about 20 minutes. The hot rolling was performed on a two-high mill having 135 mm diameter rolls and rotating at a speed of 72 r.p.m. A covered view of the preheating and hot rolling arrangement has been shown in Fig. 3.3. It can be seen that the extended exit zone of the preheating chamber kept the sponge iron block under protective atmosphere upto the nip of the rolls. The standard procedure used for hot rolling was as follows

(1) A small hole was drilled near one edge of the sponge iron block and a nichrome wire of 20 gauge was tied to it.

(2) The sponge iron block was pushed into the hot zone of the reheating chamber.

(3) The preheating furnace was placed in front of the rolling mill, so that the extended exit was very close to the rolls.

(4) The roll gap was adjusted to the required level and the rolling mill was switched on.

(5) The heated sponge iron block was now pulled in between the rotating rolls with the help of the attached wire.

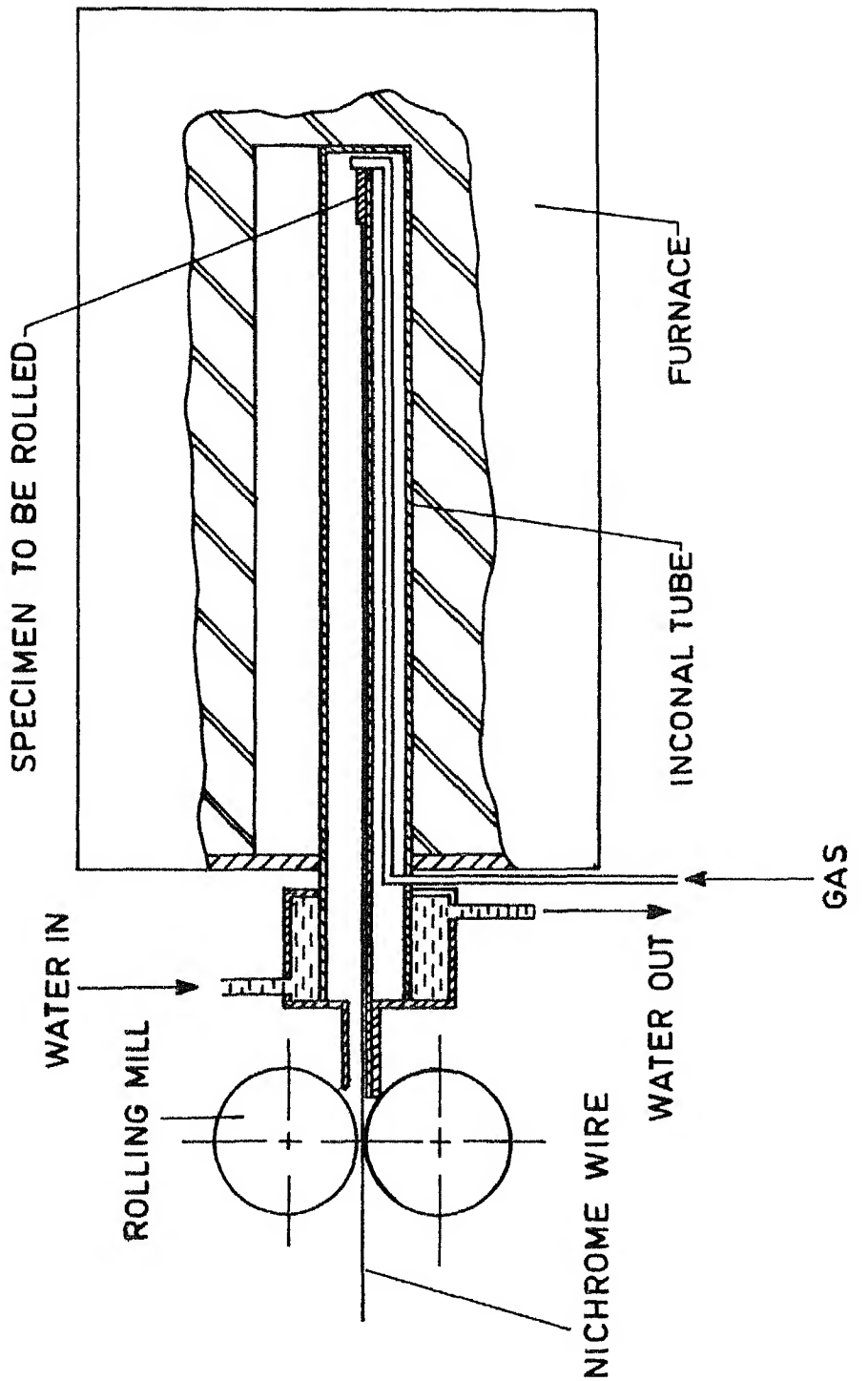

Fig 3 3 Hot rolling arrangement

(6) The hot rolled strip coming out of the mill was quickly placed in a charcoal bed to prevent oxidation.

About 80% thickness deformation is needed in order to produce fully dense strip. The hot rolling of sponge iron block into fully dense strip could not be done in a single pass as the rolling mill could not grip the feed. Hence full densification by hot rolling was achieved in two passes. The thickness of the hot rolled strip after the first pass was about 3 mm and after the second pass it was about 1 mm. It was subsequently hot rolled to about 0.6 mm thickness after trimming the edges. The hot rolled strip was subsequently annealed at 700°C for 30 minutes in a hydrogen atmosphere. This also reduced the scales formed on the surface.

3.2.4 Cold Rolling and Annealing of the Hot Rolled and Annealed Direct Strip

The cold rolling of the hot rolled strips was done on the same rolling mill, but using different rolls. Machine oil was used as a lubricant for cold rolling. The direction of cold rolling was parallel to that of the hot rolling in all the cases. The percentage thickness reduction given by cold rolling was 40,50,60,70 and 80% . Subsequently the strips were annealed for 90 minutes in a hydrogen atmosphere.

3.2.5 Mechanical Testing

Load versus elongation curves were drawn on Instron Universal Testing Machine at a crosshead speed of 0.5 mm/min,

from which U.T.S. and Y.S. were determined. The percentage elongation was determined by measuring the seperation between two marks, put on the specimen, before and after the mechanical testing. All the mechanical testing was done at room temperature. Because of the shortage of strip material, the size of the specimen used for mechanical testing, as shown in Fig.3.4 was not according to the BS 18 specification for steel strips and sheets. However the geometry of the specimen was maintained according to the above specification. Four specimens were tested in each case.

3.2.6 Carbon Analysis

For carbon analysis, the samples were taken from three different areas, namely, corner, edge and Centre (Fig. 3.5) of the blocks reduced at 1150°C for three different periods of time - 1.5 hours, 3 hours and 6 hours. For each set three samples were taken and were sent to three different outside agencies to ensure the correctness of the result. These agencies have determined the carbon content of the samples by Leco Carbon determination method.

A great care has to be taken in taking the samples from the block. As the gradient of carbon concentration is very sharp at the corner and edges a very thin sample should be taken from these areas to get the correct carbon content in these areas, and, at the same time, the thickness of the cut samples sent to three agencies should be same otherwise

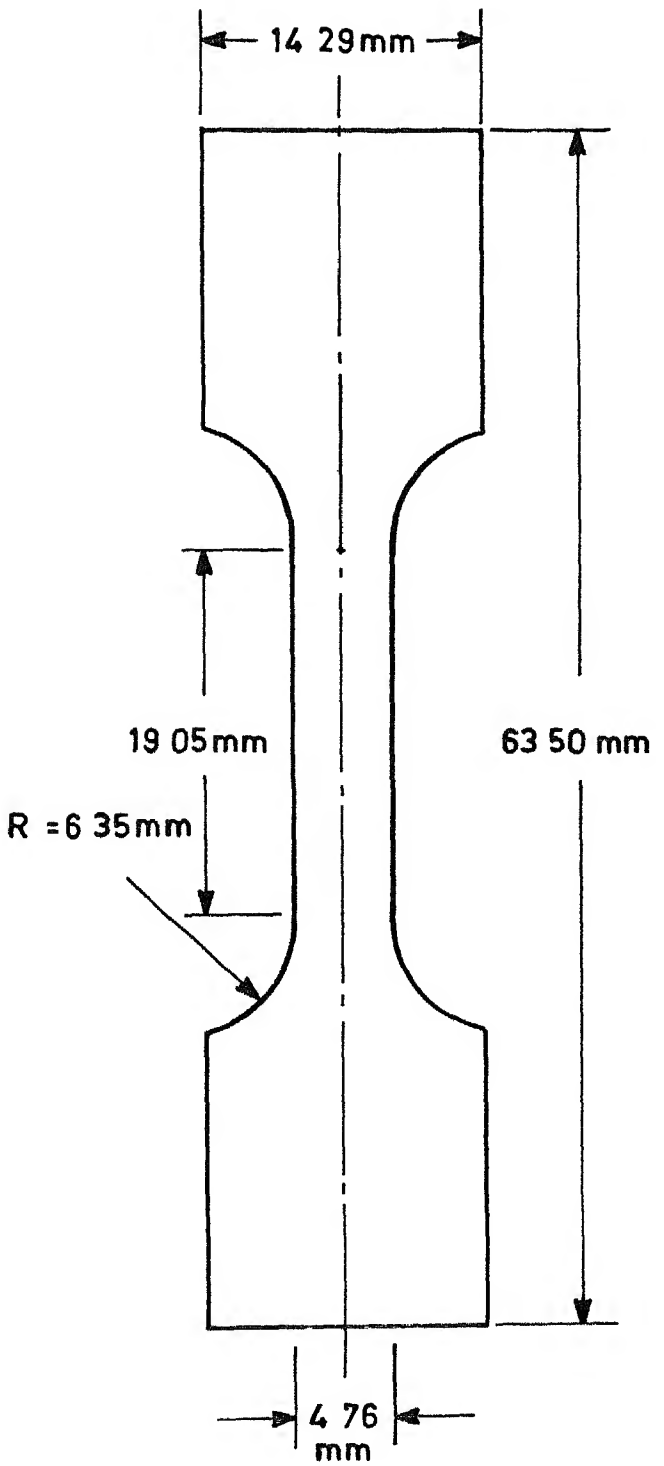

Fig 3 4 Tensile test specimen.

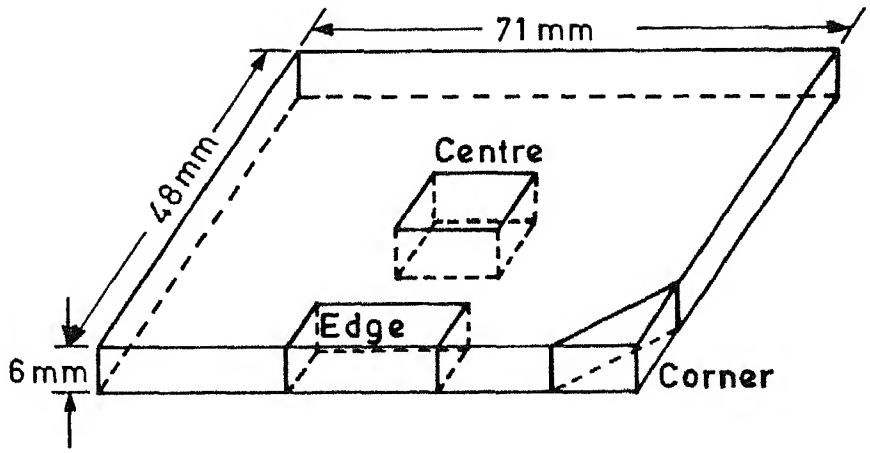

Fig 3 5 The corner, edge and centre areas of the block

there are likely to be variations in the carbon content reported by different agencies for the same set. The centre area does not pose great problem in sample collection as the carbon gradient is not so sharp in this region.

CHAPTER 4

REDUCTION OF MAGNETITE SUPERCONCENTRATE BLOCK IN A PACKED BED OF SAW DUST CHARCOAL

Vethanayagam[12], in his study, found conventional charcoal and devolatalized brown coal to be suitable to be used as reductants for Magnetite superconcentrate block from the viewpoints of absence of sticking of coal mass to the tube wall, easy availability, good reduction behaviour, etc.. However, there was one problem encountered with these reductants. The fine size fraction of these reductants left behind a few clinkers on the surface of the reduced block, while using coarse size coal, say > 10mm, there was a problem of reoxidation of reduced block. Mainly, to solve this problem and to obtain a smooth surface finish of reduced block, the saw dust charcoal was tried as a reductant.

4.1 Saw Dust Charcoal and Surface Finish of the Reduced Block.

Saw dust charcoal was fine, equi-sized and rice grain shaped in physical appearence. A scanning electron microscope photograph of saw dust charcoal particles has been shown in the Fig.4.1. The Sieve analysis of saw dust charcoal is also given in Table 4.1. With this size of coal, there was no question of reoxidation of the reduced block. Saw dust charcoal provided an uniform hydrostatic pressure to give an uniform shape to the reduced block. It did not stick to the surface of the reduced block and the problem of formation of clinkers on the surface of the reduced block was avoided.

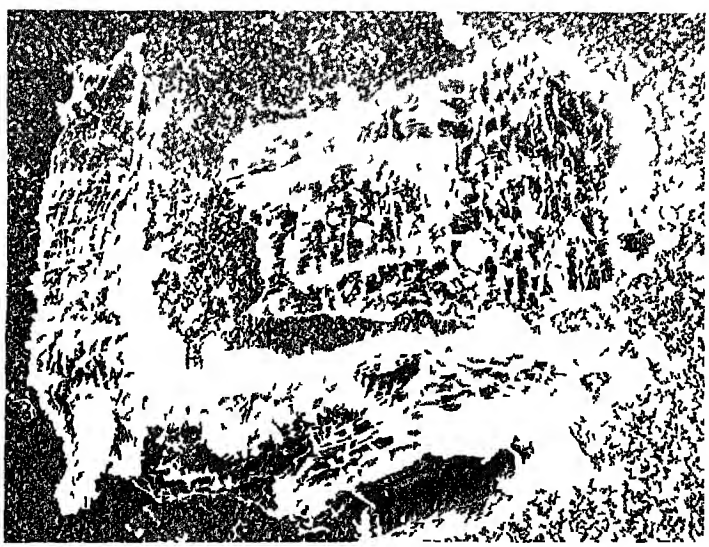

Fig. 4.1: A View of Saw Dust Charcoal Particles (100X)

Table 4.1 Sieve Analysis of Saw Dust Charcoal

S.No.	Sieve Size Mesh (mm)	Wt. %
1	+ 6 (+ 3.327)	0
2	- 6 + 20 (- 3.327 + 0.833)	3.4
3	- 20 + 28 (- 0.833 + 0.589)	9.5
4	- 28 + 35 (- 0.589 + 0.417)	13.4
5	- 35 + 48 (-0.417 + 0.295)	30.8
6	- 48 + 65 (-0.295 + 0.208)	16.8
7	- 65 + 100 (-0.208 + 0.149)	8.7
8	- 100 (- 0.149)	14.4

The surface finish of the reduced block was quite good. A view of the reduced sponge iron block has been shown in Fig. 4.2

4.2 Reducibility of Saw Dust Charcoal :

Fig. 4.3 shows the percent weight loss in the iron oxide superconcentrate block as a function of time at a reduction temperature of 1150°C, using saw dust charcoal as a reductant. The above figure also shows the weight loss behaviour at the same temperature using devolatalised brown coal and conventional charcoal as reductants. It can be seen that saw dust charcoal is slightly inferior to conventional charcoal and devolatalised brown coal in reducibility. Nevertheless, the entire block was almost completely reduced in one and half hour. To ensure that the last traces of iron oxide have been completely reduced, the blocks reduced for 3 hours have been used for the subsequent study.

4.3 Discussion

Saw dust charcoal has all the properties to be used as a reductant for magnetite superconcentrate block. It is cheap and readily available, does not stick to the wall, and does not deteriorate the shape and surface finish of the reduced block.

The reducibility of saw dust charcoal is not as good as that of the devolatilized brown coal or conventional charcoal but it is good enough not to create any problem regarding the total time taken for reduction.

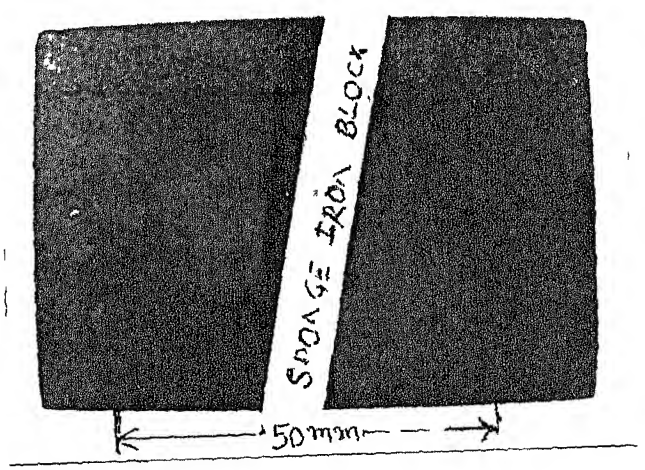

Fig. 4.2: A View of Peduced Sponge Iron Block

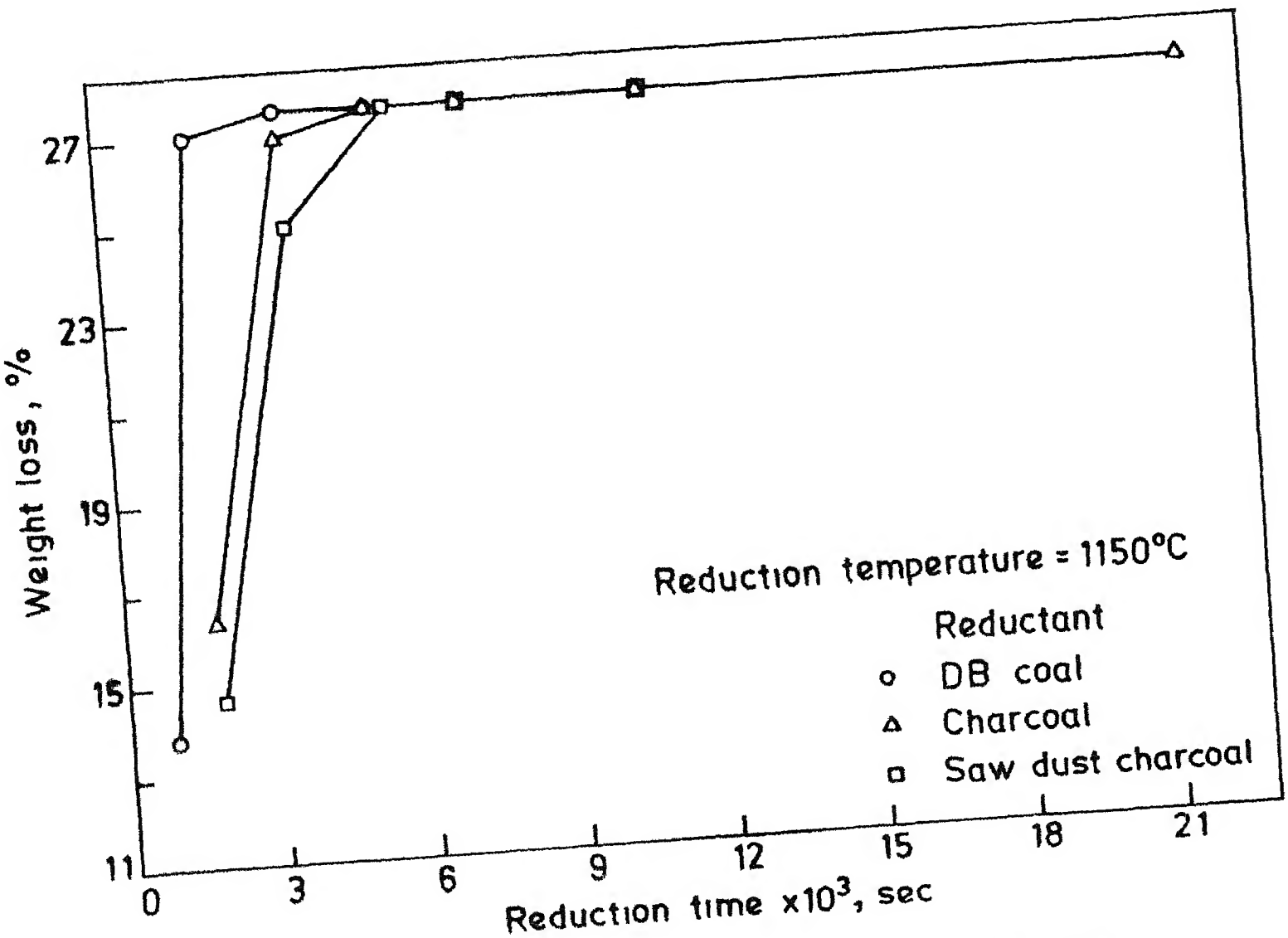

Fig 4.3 Reduction time Vs % weight loss

The reducibility of a coal depends on the activity of carbon in the coal particles, that is, in turn, depends on the chemical composition, shape, size, surface properties and the porosity level present in the coal particles. A study can be taken as to look into why the reducibility of saw dust charcoal is less than the reducibility of conventional charcoal which is less than the reducibility of devolatalized brown coal. Coconut charcoal can also be tried as a reductant as it is expected to have other properties same as saw dust charcoal but it has been reported to have higher reducibility for the reduction of iron oxides, by some workers[13].

CHAPTER 5

CARBURIZATION OF MAGNETITE SUPERCONCENTRATE BLOCK IN PACKED SAW DUST CHARCOAL BED AND DECARBURIZATION OF THE BLOCK DURING PREHEATING IN HYDROGEN ATMOSPHERE PRIOR TO HOT ROLLING

Magnetite superconcentrate green compacts were kept inside a ruffle furnace packed with saw dust charcoal at 1150°C for 3 hours to ensure the complete reduction of iron oxide. But the conditions are similar to packed bed carburization and simultaneous carburization of the block which is being reduced is also expected to take place. Therefore the carbon analysis of reduced sponge iron block as well as the hot rolled strips was carried out.

The variation of carbon content at corner, edge and centre of the sponge iron block for a fixed reduction time, at 1150°C, is shown in Fig. 5.1(a), 5.1(b) and 5.1(c). The variation of carbon content at a fixed location on sponge iron block as a function of reduction time at 1150°C is shown in Fig. 5.2(a), 5.2(b), 5.2(c) for corner, edge and centre area respectively. The corner, edge and centre areas of the block have been shown in Fig. 5.3. These datas have also been shown in Table 5.1 which also contains the carbon content of the hot rolled strip at the edge and centre areas.

It is clear from the high carbon levels shown in Table 5.1 that simultaneous carburization is taking place during the reduction of the magnetite superconcentrate block. But carbon level is reduced to a very low level in the hot rolled strip

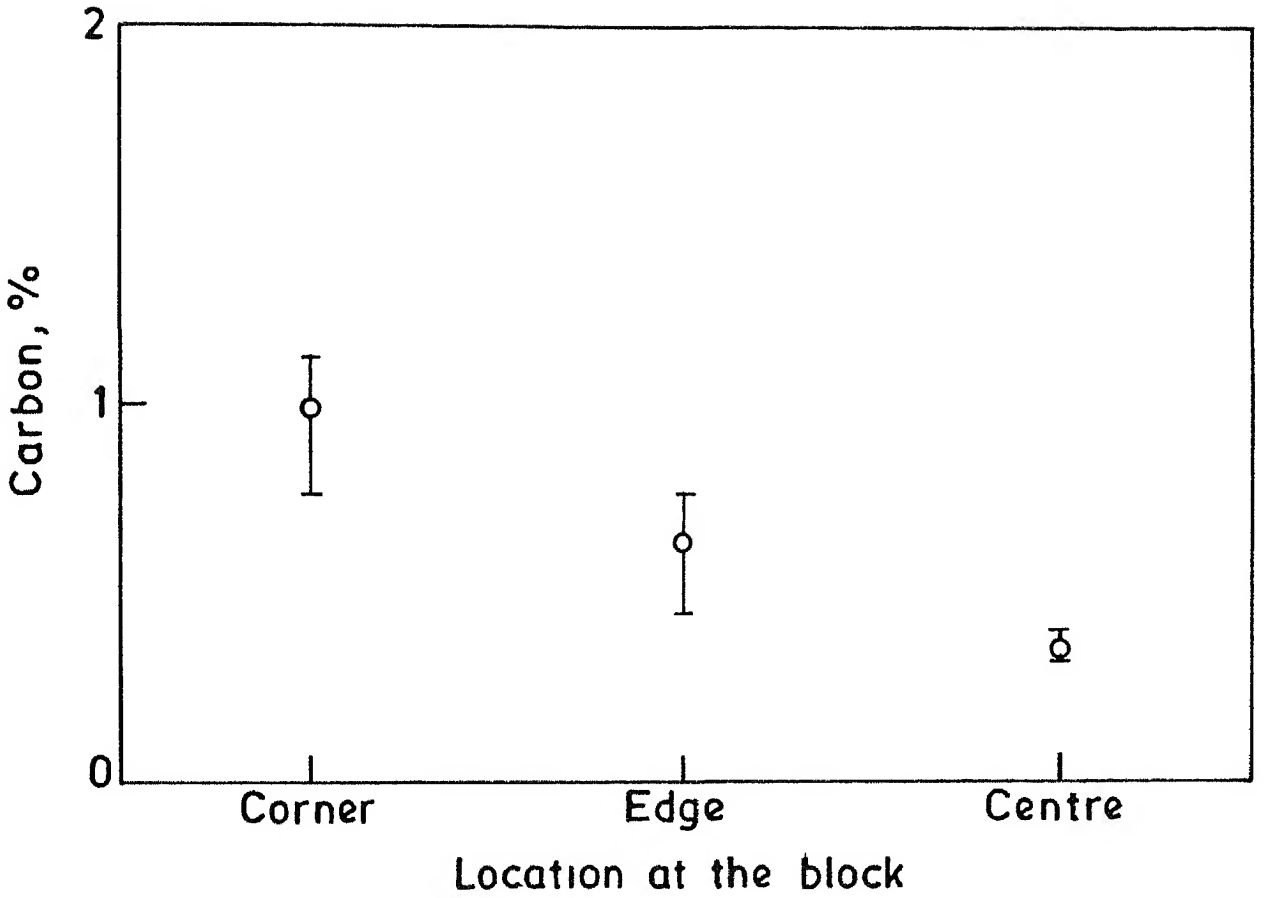

Fig 5 1(a) Carbon content at the corner, edge and centre areas of the block reduced at 1150°C for 15 hours

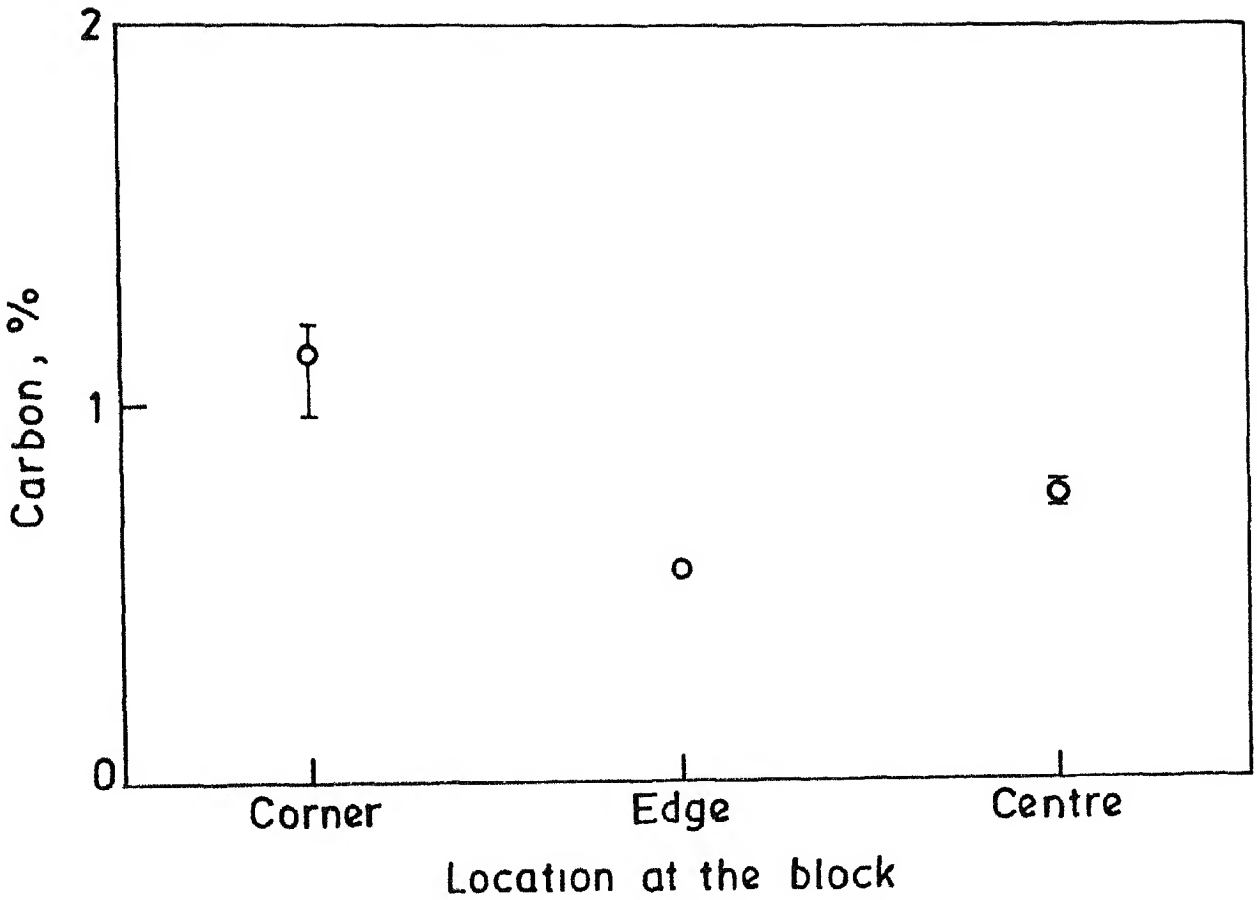

Fig 5 1(b) Carbon content at the corner, edge & centre areas of the block reduced at 1150°C for 3 hours

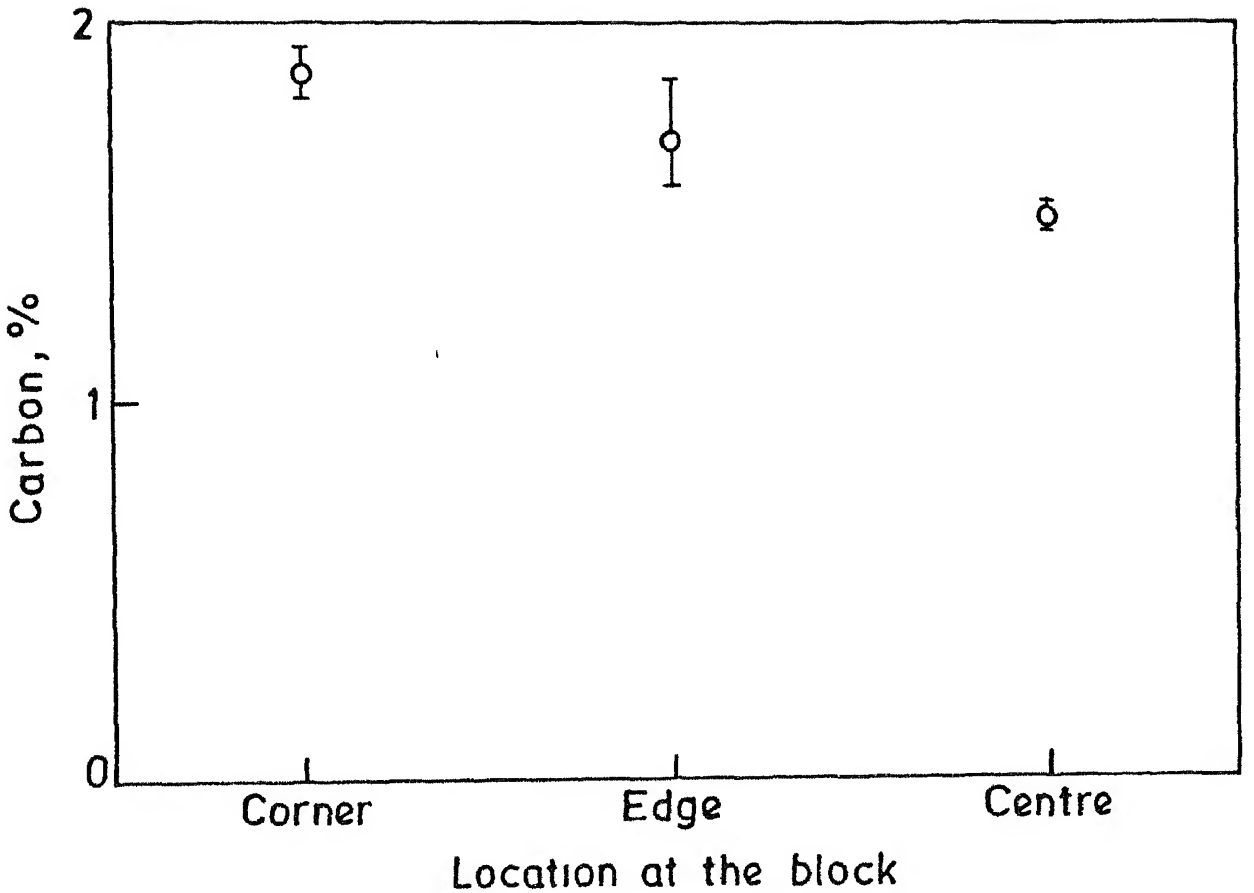

Fig 5 1(c) Carbon content at the corner, edge & centre areas of the block reduced at 1150°C for 6 hours

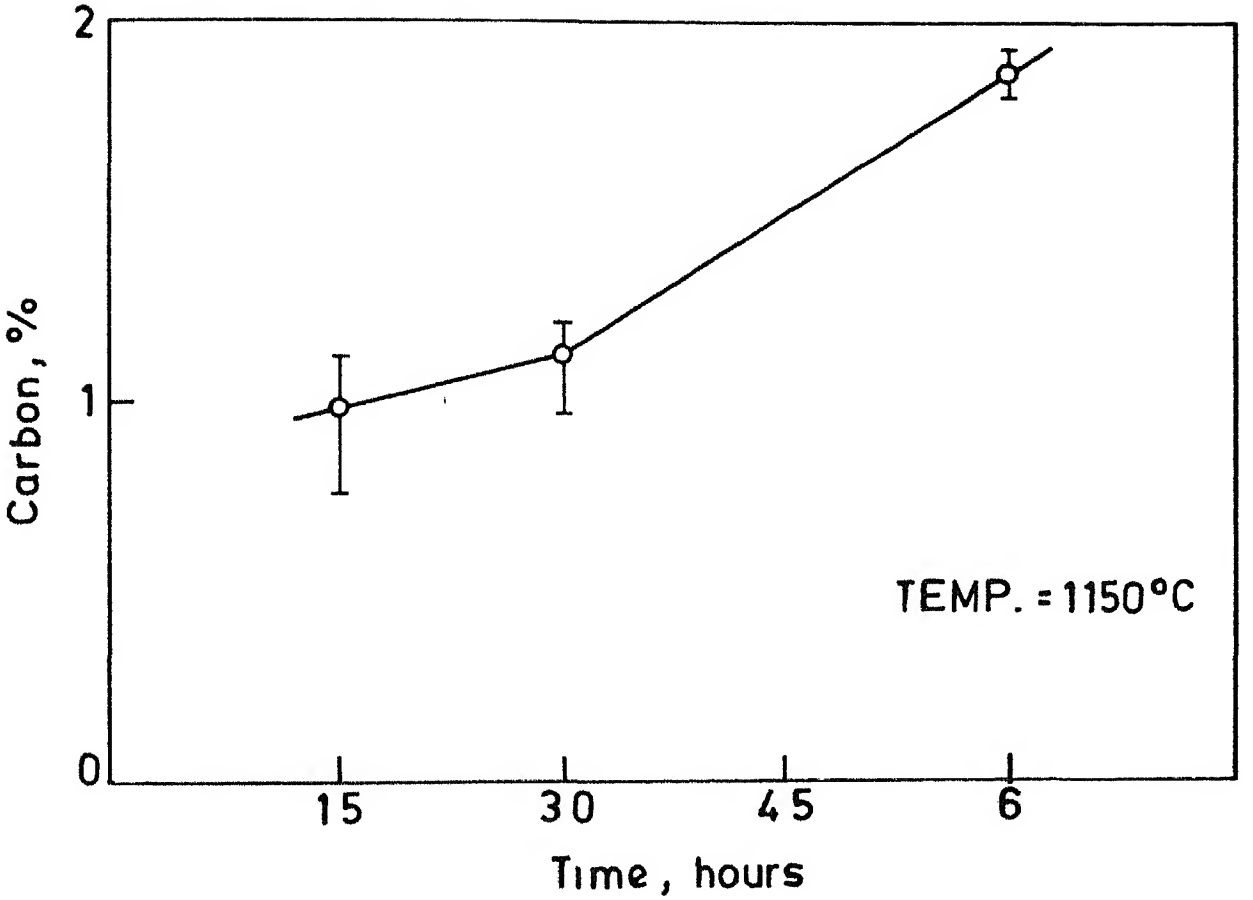

Fig 5 2(a) Variation of carbon content at the corner area of the block as a function of reduction time

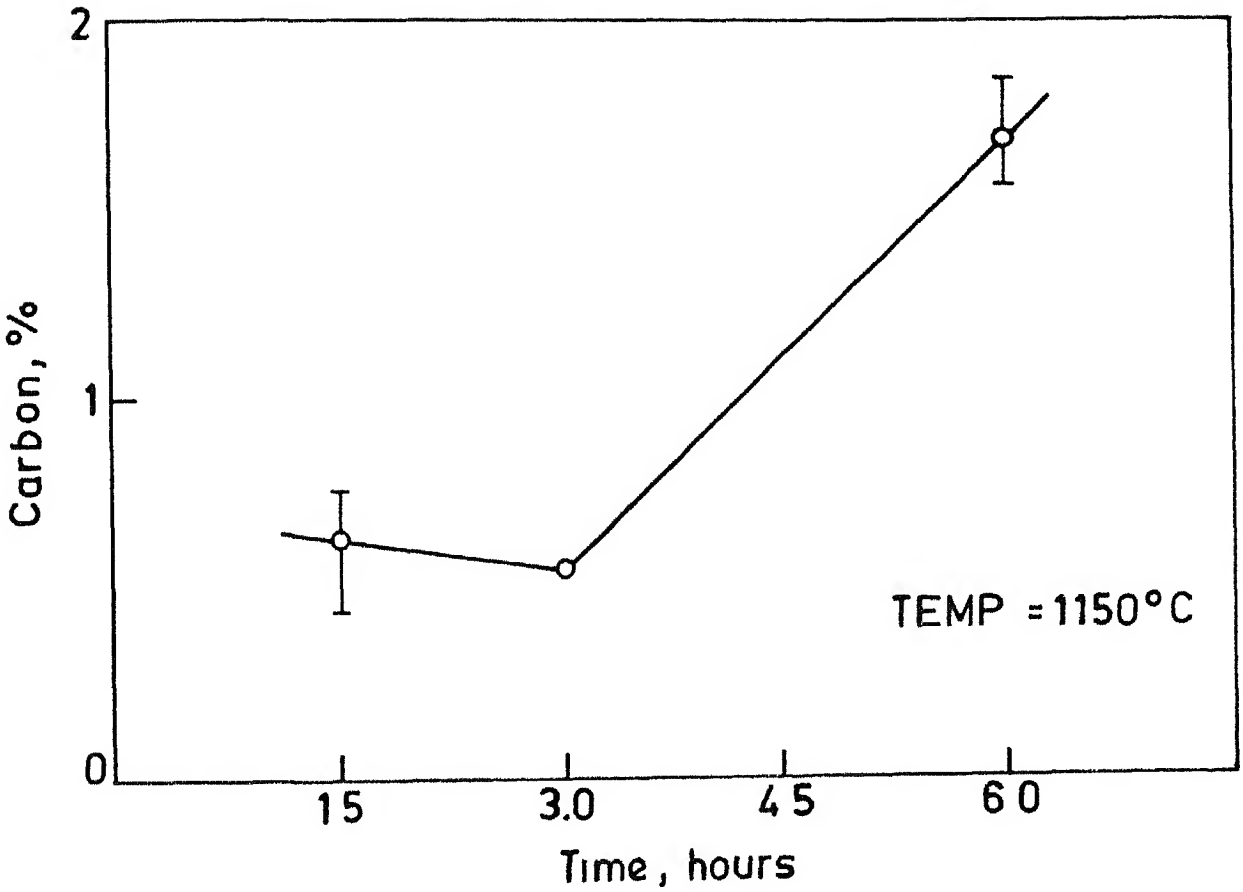

Fig 5 2(b) Variation of carbon content at the edge area of the block as a function of reduction time

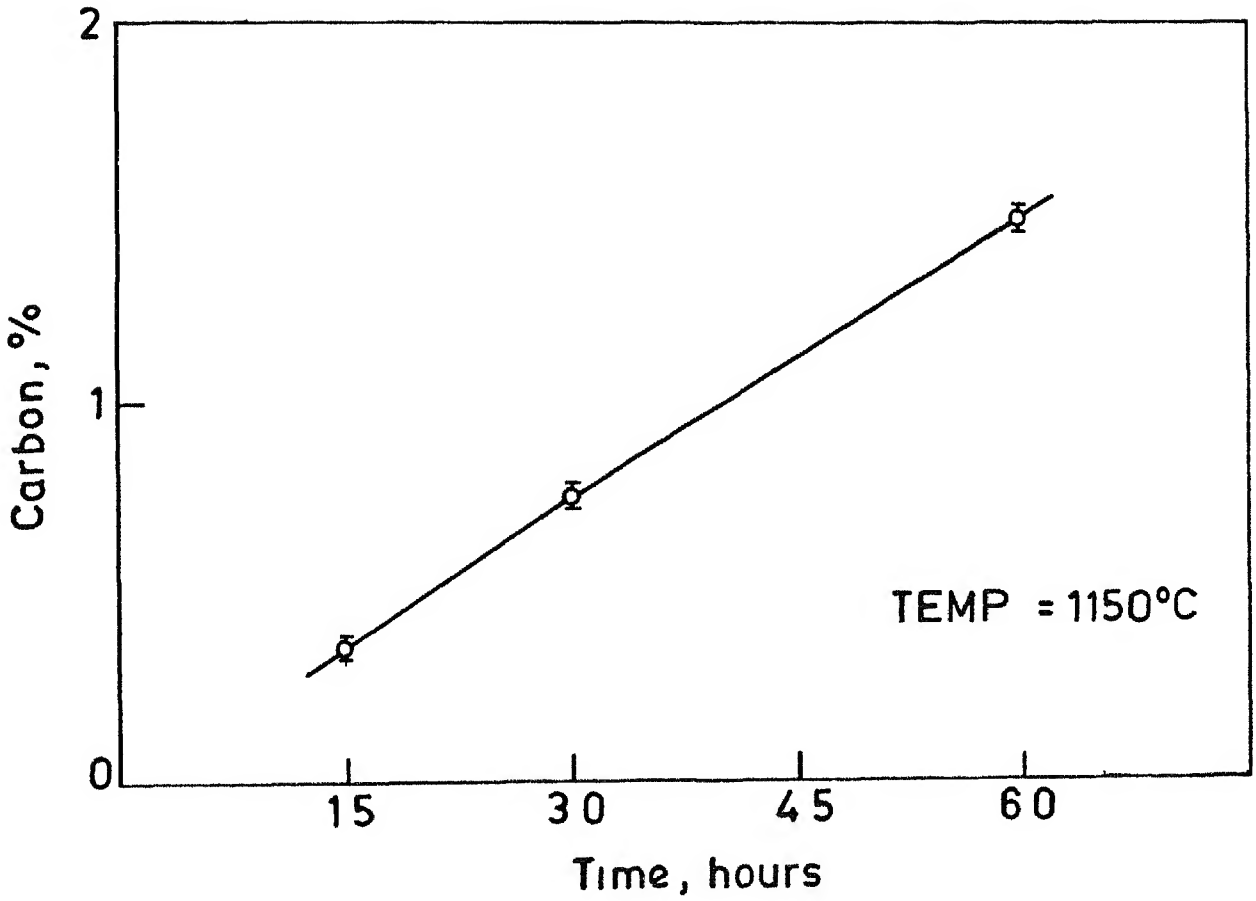

Fig 5 2(c) Variation of carbon content at the centre area of the block as a function of reduction time

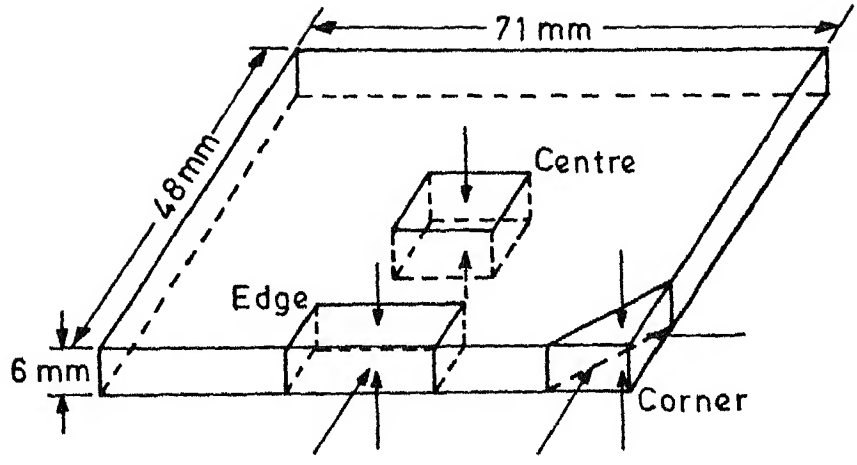

Fig 5 3 Carbon diffusion in the magnetite super-concentrate block at the corner, edge and centre areas (arrows show the direction of carbon diffusion)

Table 5.1 . Carbon Analysis of Sponge Iron Block Reduced at 1150°c

Time of reduction (hours)	Carbon content (wt %)											
	Corner				Edge				Centre			
	Agency (1) (x_1)	Agency (2) (x_2)	Agency (3) (x_3)	Mean $\frac{x_1+x_2+x_3}{3}$	Agency (1) (x_4)	Agency (2) (x_5)	Agency (3) (x_6)	Mean $\frac{x_4+x_5+x_6}{3}$	Agency (1) (x_7)	Agency (2) (x_8)	Agency (3) (x_9)	Mean $\frac{x_7+x_8+x_9}{3}$
1.5	1.09	1.12	0.76	0.99	0.69	0.76	0.44	0.63	0.33	0.33	0.39	0.35
3.0	1.20	0.97	1.21	1.13	x	0.56	0.55	0.56	0.79	0.73	0.73	0.75
6.0	1.81	1.86	1.94	1.87	1.85	1.57	1.65	1.69	1.46	1.52	x	1.49
Caron content (wt %) of hot rolled strip produced from sponge iron block reduced at 1150°c for 3 hours and preheated in hydrogen atmosphere for 20 minutes.					0.08	x	0.06	0.07	0.07	0.04	0.05	0.05

'x' data not available

which means that the process of decarburization is also taking place at a stage in between the reduced sponge iron block and hot rolled strips, i.e., during the preheating in hydrogen atmosphere prior to hot rolling.

5.1 Discussion

5.1.1 Carburization

Although there are few variations in the carbon analysis done by the different agencies (Fig. 5.1 (a), 5.1 (b) & 5.1 (c)), it is apparent that, by and large, the carbon content is maximum at the corner area, minimum at the centre area, and in between the two at the edge area of the block. The variations in the carbon analysis may be due to difficulties in taking the samples from the block for carbon analysis, as explained in section 3.2.6, or due to experimental errors in carbon analysis. This is quite expected as at corners, the carbon diffuses from four sides, at edges, it diffuses from three sides and at the centre, from two sides only, as shown in Fig. 5.3.

Fig. 5.2 (a), 5.2 (b) and 5.2 (c) show that the carbon content, at a fixed location at a fixed temperature, increases with the time of reduction. This can also be explained on the basis of diffusion of carbon. If the time for diffusion of carbon is more, the carbon content inside the block will also be more.

The mechanism of carburization of steel may be understood on the basis of two fundamental concepts

(1) source supplying the carbon and the transfer of carbon to the steel surface, and

(2) a diffusion, which is concerned with the movement of carbon in steel itself, and is influenced by the properties of the steel.

To understand the various reactions involved in the process of carburization, it will be more convenient to deal reduction and carburization simultaneously as carburization, in the present case, is preceded by the reduction of iron oxides.

The first step in the reduction of magnetite is the formation of CO which is formed in two steps. At lower temperatures, first CO_2 is formed

$$C_{(s)} + O_2(g) \text{ ------ } CO_2(g) \text{ -- (1)}$$

This CO_2 then reacts with carbon at high temperatures to forms CO:

$$C_{(s)} + CO_2(g) \text{ -------- } 2CO(g) \text{ ----(2)}$$

The oxygen for the above reaction comes from the air encrapped in the pores of packed bed of saw dust charcoal as well as the magnetite superconcentrate block.

CO is also generated at the point contacts between the iron oxides and carbon, i.e.,

$$Fe_3O_4(s) + C(s) \text{ ----- } 3\ FeC(s) + CO\ (g) \text{ ----- } (3)$$

$$FeO(s) + C(s) \text{ ----- } Fe(s) + CO\ (g) \text{ ----- } (4)$$

The CO formed from these reactions then reacts with the iron oxides to form CO_2

$$Fe_3O_4(s) + CO\ (g) \text{ ----- } 3\ FeO(s) + CO_2(g) \text{ ----- } (5)$$

$$FeO(s) + CO\ (g) \text{ ----- } Fe(s) + CO_2\ (g) \text{ ----- } (6)$$

The CO_2 then reacts with the excess carbon to regenerate CO

$$C(s) + CO_2(g) \text{ ----- } 2\ CO(g) \text{ ----- } (7)$$

and the cycle is repeated.

Once the reduction of a layer is complete, it starts getting carburized. First the nascent carbon is formed at the surface

$$2CO(g) \text{ ----- } CO_2(g) + C \text{ ----- } (8)$$

then it is this nascent carbon that diffuses inside the block. CO_2 reacts with the excess carbon to regenerate CO

$$CO_2(g) + C(s) \text{ ----- } 2\ CO(g) \text{ ----- } (9)$$

and the cycle is repeated.

The fundamental law governing the diffusion of carbon in steel is the Fick's law of diffusion

$$\frac{dm}{dt} = -SD\frac{dc}{dx}$$

where $\frac{dm}{dt}$ may be taken as the time rate of flow of the solute carbon through the area s. D is diffusion constant

and $-\frac{dC}{dx}$ is the rate at which the carbon concentration C varies with the depth x. The flow of carbon in γ-steel is, in part, analogous to the flow of heat in solids, and to some extent expression developed for the conduction of heat are applicable.

Foster[14], after carburizing compacts made from carbonyl iron powder of 8 μm average particle size concluded that a density of at least 6.85 g/cc is necessary to give a well defined case, i.e. to cut the diffusion path of carbon. The key requirement was stated to be a fully recrystallized structure containing isolated pores away from grain boundaries. The porous sponge iron block, having a density in the range of 1.7-2.5 g/cc and porosity level of 70-80%, has interconnected pores. Hence the contact area S, in the present case, will be much more as compared to a steel block of same size, and carbon will diffuse to a greater depth. The mechanisms involved in carbon diffusion through a porous iron mass are, however, are not yet fully understood but the presence of interconnected porosity, allowing gas to permeate, is obviously the major reason for deep penetration of carbon during the carburization process.

5.1.2 Decarburization

When a carbon steel is heated in pure hydrogen it has been suggested[15,16] that direct reaction may take place between the gas and the carbon in the steel .

$$[C]_{Fe} + 2H_2(g) \text{ ---- } CH_4(g) \text{ ---------- (10)}$$

The above mechanism seems to be most unlikely in view of unstability of CH_4 at high temperature. The experimental observations also show that the rate of decarburization of sheets in pure hydrogen is very less. Johansson and Von Seth[17], after heating plain carbon steel at 1150°C for 1f hours found that carbon content came down to 0.22% from a level of 0.72% and to 0.38% from a level of 1.13% . The decarburization of porous sponge iron block in pure hydrogen atmosphere will be some-what faster due to larger contact area between the gas and the block owing to the presence of interconnected pores but it can not explain such a fast decarburization rate that reduces the carbon level from about 1.2% to around 0.05% in 20 minutes at a temperature of 1150°C. So some other mechanism has to be found.

Water vapour increases considerably the decarburizing power of otherwise pure hydrogen. Austin[18] has supplied data on the decarburisation at 800°C of a steel containing 1.07% carbon and 1.42% Chromium (Fig. 5.4). His results indicate that whereas pure hydrogen has a real, but limited, decarburizing action, the presence of water vapour, at least in concentrations above 10 milligrams per cubic feet (0.37 ppm), gives rise to very pronounced decarburization. The nature of the reactions involved is not fully understood, but it is probable that the process is a two stage one[19], in the first of which the water vapour is dissociated, setting free nascent

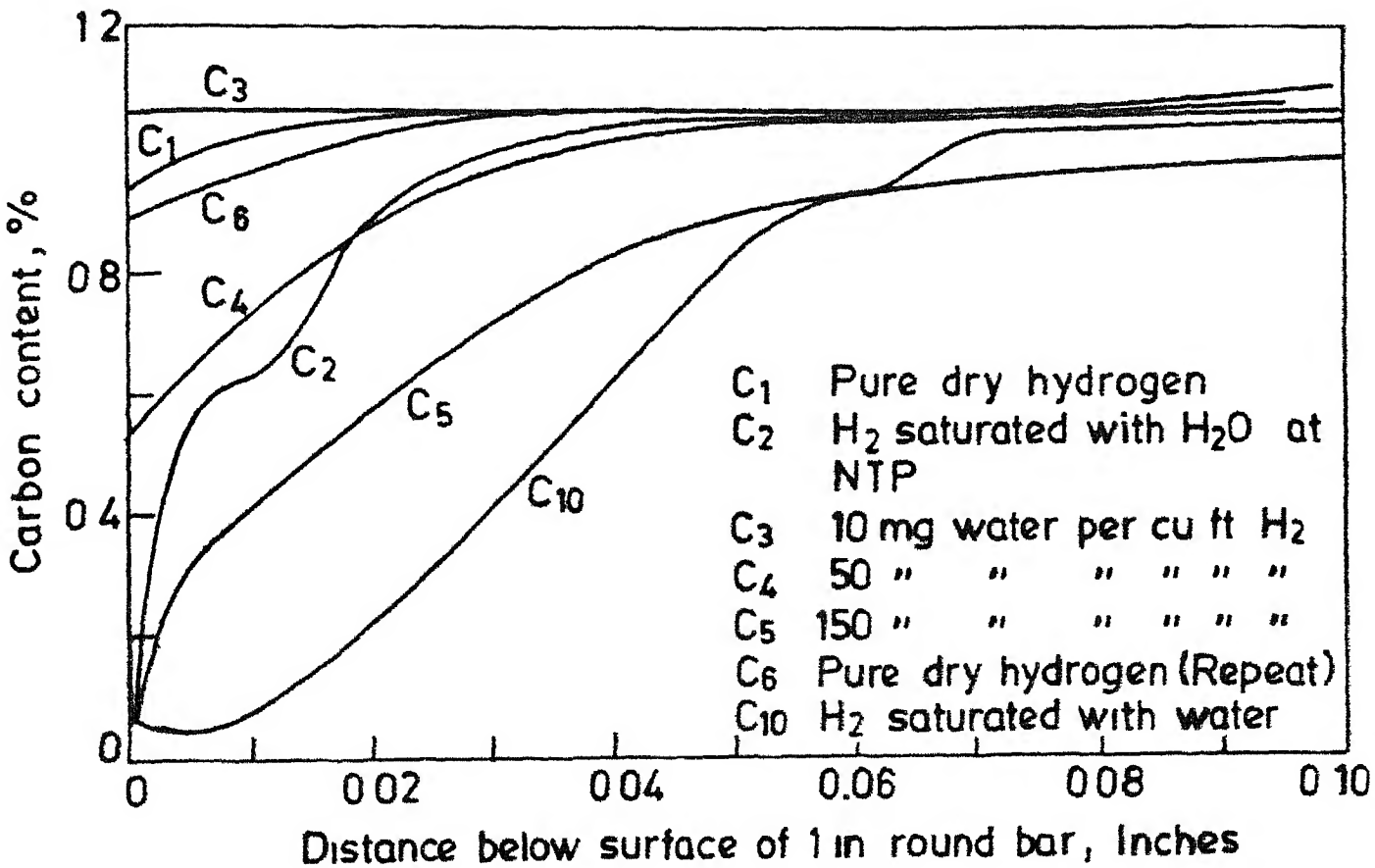

Fig 5 4 Decarburisation of 1 07 % Carbon, 1 42 % chromium steel in dry and moist hydrogen C_2 treated 55 hours at 750°C ; C_1, C_3, C_4, C_5, C_6, C_{10} treated 55 hours at 800°C [18]

hydrogen and oxygen which then react with the carbon in solution in the steel,

$$H_2O \text{ -------- } H_2 + O \text{ ------} \quad (11)$$

$$[C]_{Fe} + 4\,H \text{ -- } CH_4 \quad \text{--------} \quad (12)$$

$$[C]_{Fe} + O \text{ --- } CO \quad \text{--------} \quad (13)$$

Reaction (12) and reaction (13) operate in a parallel combination and the rate of reaction will be determined by the faster step. The reaction (13) is more likely to take place in view of high stability of CO at high temperature. Hence a combination of reaction (11) & reaction (13) is, perhaps, the main mechanism, responsible for decarburization of the sponge iron block prior to hot rolling. The composition of IOLAR 3 Grade hydrogen, used in the present study, as shown in Table 3.2, does, indeed, reflect that the traces of not only water vapour but also of oxygen and carbon dioxide are present in sufficient amount to cause vigorous decarburization of the sponge iron block.

OPTIMIZATION OF COLD ROLLING DEFORMATION

In producing cold-rolled sheet from low-carbon steel, particular attention is given to the selection of the optimum degree of deformation in cold rolling. For different types of steel different amounts of cold rolling have been found optimum. At many plants, the overall reduction during cold rolling usually amounts to 50-70% [20]. It was felt necessary to optimize the amount of cold rolling for the direct steel strip produced by this new route also.

Table 6.1 & Table 6.2 show the mechanical properties of hot rolled and cold rolled annealed direct strips respectively. Fig. 6.1 , 6.2 & 6.3 show the variation of ultimate tensile strength, yield strength and percentage elongation respectively with the percentage reduction in thickness during cold rolling. From these, curves, it is clear that the range of 50 to 60% reduction in thickness during cold rolling gives the best mechanical properties.

6.1 Discussion

A characteristic feature of the direct steel strip produced from magnetite superconcentrate blocks is the presence of inclusions . Magnetite superconcentrate contains about 2 vol.% of impurities which mainly consists of oxides of Calcium, Magnesium, Aluminium and Silicon [21] . These impurities remain inside the finished strip in the form of fine inclusions distributed uniformly throughout the matrix. It is, perhaps, mainly

Table 6.1 · Mechanical Properties of Hot Rolled and Annealed Direct Strips

U.T.S. (MNm^{-2})	Y.S. ($MN\ m^{-2}$)	Elongation ($MN\ m^{-2}$)
248 ± 0.4%	160 ± 1%	34 ± 0%

Table 6.2 . Mechanical Properties of Cold Rolled and Annealed Direct Strips

S.No.	Cold rolling,%	U.T.S. ($MN\ m^{-2}$)	Y.S.,0.2, Proof Stress ($MN\ m^{-2}$)	Elongation %
1.	40	224 ± 0.5%	48 ± 4%	30 ± 0%
2.	50	240 ± 0.4%	70 ± 1.5%	37 ± 0%
3.	60	250 ± 0.4%	98 ± 4%	38 ± 5%
4.	70	231 ± 0.5%	63 ± 4.5%	30 ± 0%
5.	80	228 ± 1.0%	58 ± 5 %	30 ± 0%

Note. The limits with ± show the maximum and minimum deviation from the mean value

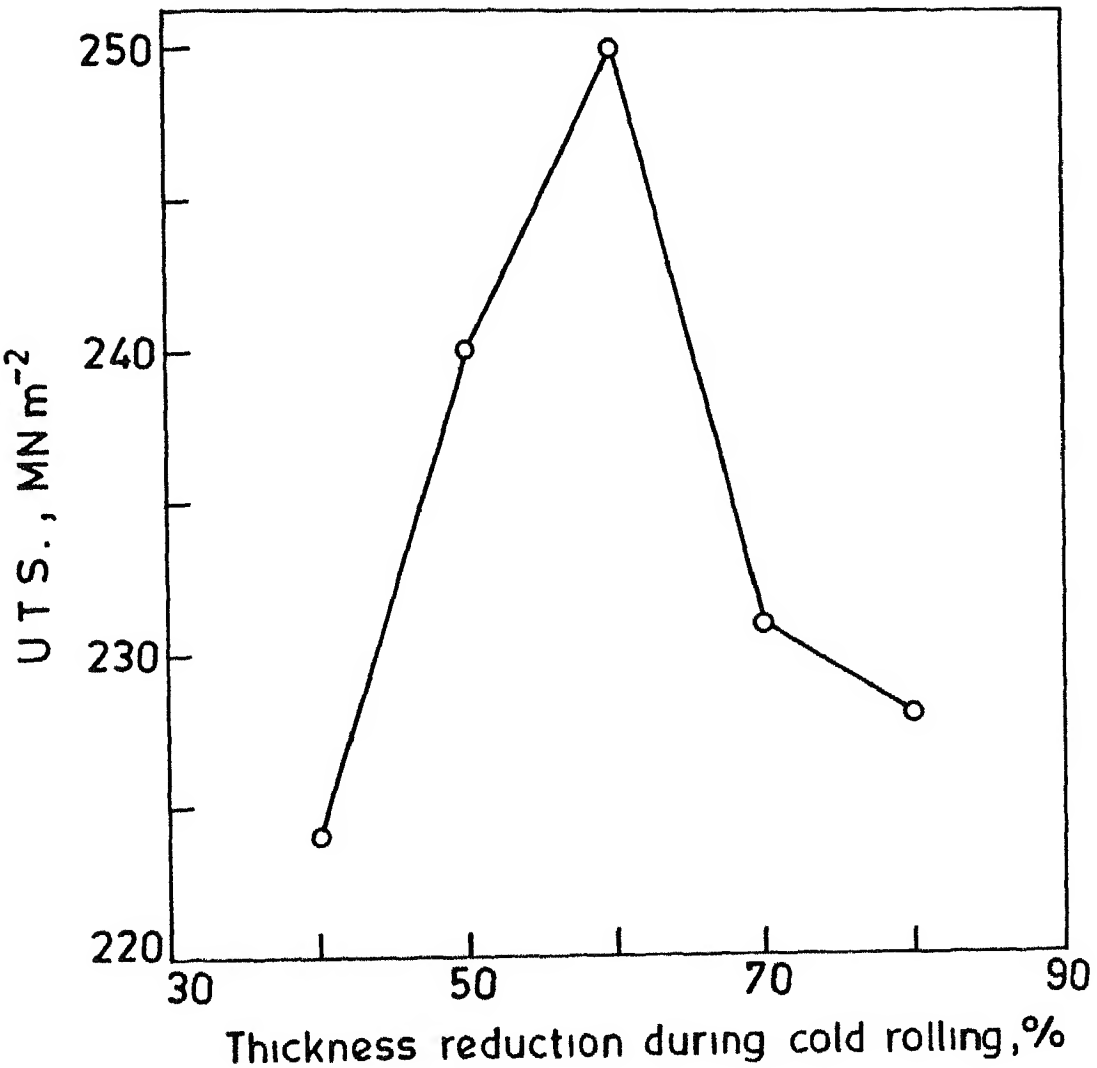

Fig 6 1 Effect of cold rolling deformation on the U T S of the annealed strip

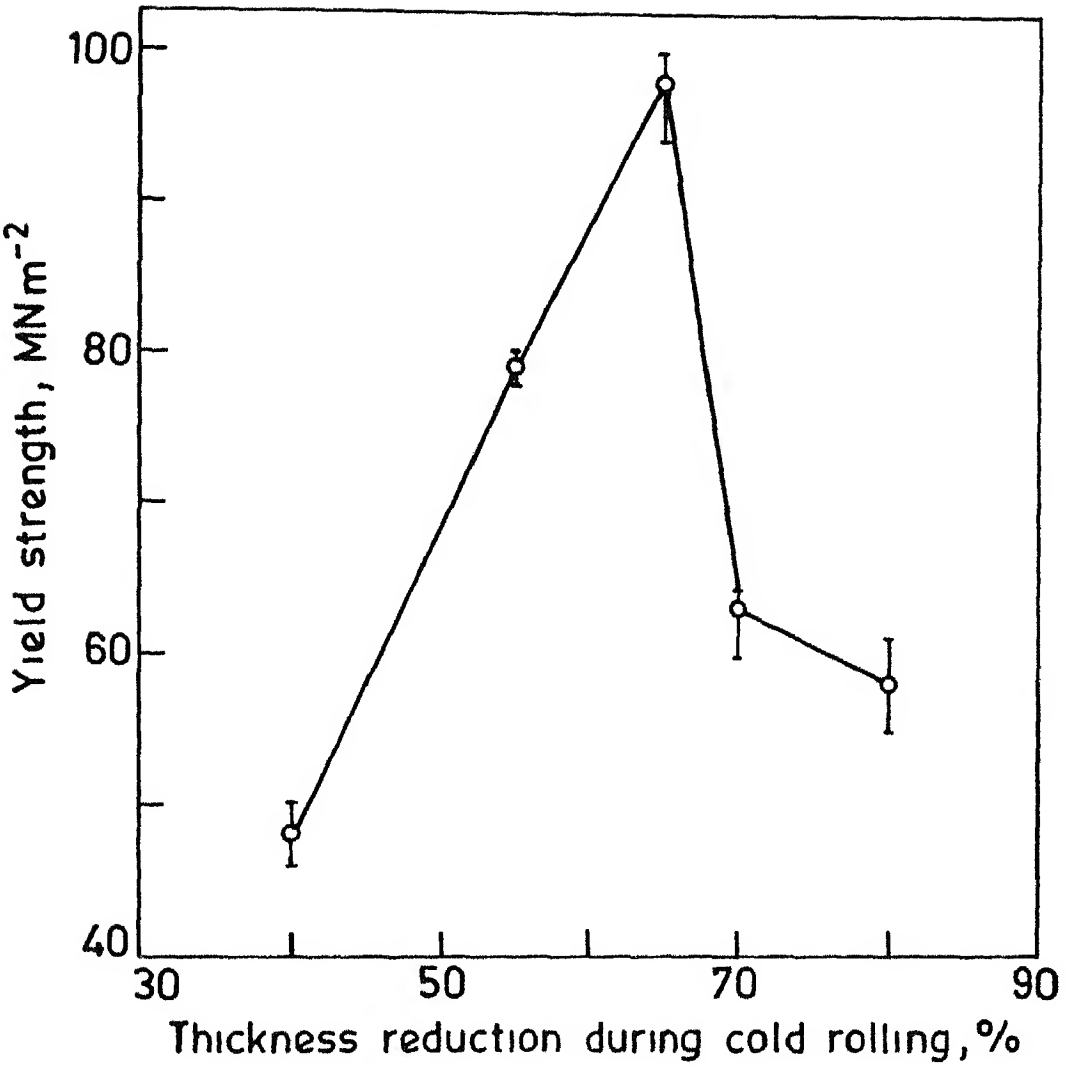

Fig 6 2 Effect of cold rolling deformation on the yield strength of the annealed strip

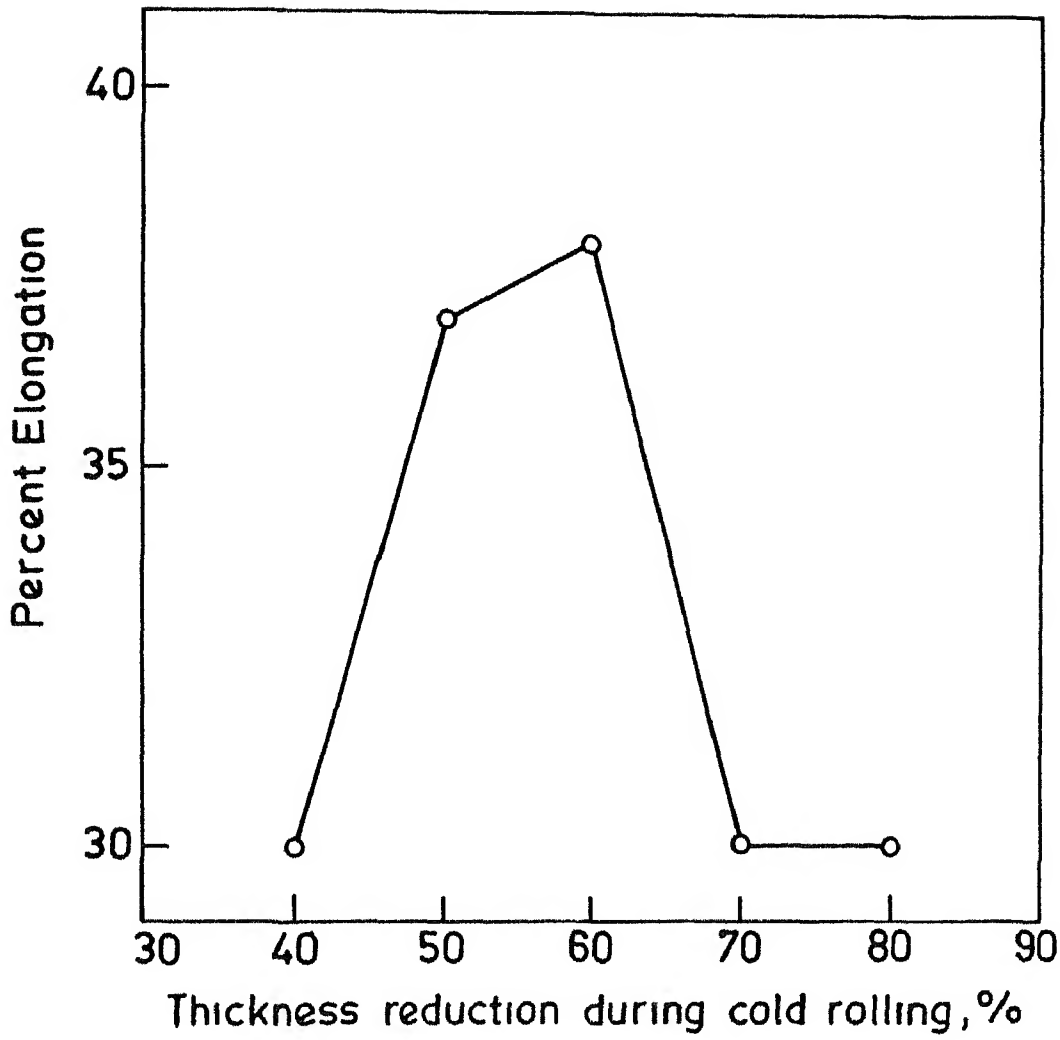

Fig 6.3 Effect of cold rolling deformation on the percent elongation of the annealed strip

these inclusions which are responsible for the variation in mechanical properties during cold rolling.

As degree of deformation increases, nucleation rate of the recrystallized nuclie considerably exceeds the growth rate during annealing resulting in a decrease in grain size. Hence, as the amount of cold rolling increases, mechanical properties of the cold rolled and annealed strip improve due to reduction in grain size. Simultaneously, stresses are generated at matrix-inclusion interfere during cold rolling and holes or micro cracks may be formed around hard inclusion which are erbedded in relatively soft steel matrix. Mechanism of hole formation and growth under tensile stress is well understood. Gurland and Plateau[22], in equating the elastic energy stored in the particle to the work of producing a particle size crack, have calculated that the applied stress required (σ) is given by

$$\sigma = q \left(\frac{E\gamma}{d}\right)^{1/2}$$

where q is a stress-concentration factor, E is Young's modulus, γ is the fracture surface energy, and d is the particle diameter. The value of q depends both on the relative rigidity of the particle and matrix and on the shape of the particle. So such cracks may form around the inclusion during cold rolling also if the required stress level is reached. The formation of these cracks will cause deterioration in mechanical properties.

The metallographic examination of micro-cracks at the matrix-inclusion interface involves careful polishing. But during polishing some inclusions may fall out, and micro-cracks may also appear at the interface of some inclusions with matrix due to decohesion. The preliminary trials of scanning electron microscope examination of the cold rolled specimens did not give a clear indication whether the micro-cracks were actually formed during the cold rolling or not. Much more detailed work on a large number of specimens is needed to ascertain this fact.

Mechanical properties improve with cold rolling due to grain refinement but after a certain amount of cold rolling, perhaps, the microcracks formation takes place around the inclusions resulting in a decline in mechanical properties of the cold rolled and annealed sheet.

CONCLUSIONS

1. It is possible to produce good quality, fully densified steel strip by 'Direct strip Process' from magnetite superconcentrate block using gacia Arabica as a binder and saw dust charcoal as a reducing agent.

2. The present investigation shows that it is possible to fully reduce the magnetite superconcentrate block of size 71 x 48 x 6 mm^3 in a packed bed of saw dust charcoal in one and half hour at 1150°C.

3. Saw dust charcoal has been found to be suitable for reducing the magnetite superconcentrate block as it gives a good surface finish to the reduced block and an extra step of cleaning the surface of reduced block prior to hot rolling is avoided.

4. Simultaneous carburization takes place during the process of reduction of magnetite superconcentrate block in packed bed of saw dust charcoal.

5. Carbon content of the sponge iron block is maximum at the corner and minimum at the centre. At 1150°C, for 3 hours of reduction time, the average carbon content is 1.13% at the corner and 0.75% at the centre.

6. Carbon content of the block increases with increase in time for which the block is kept inside the saw dust charcoal bed for reduction.

7. Decarburization of the sponge iron block takes place during preheating in hydrogen atmosphere prior to hot

rolling, reducing carbon to very low level and distributed uniformly along the width in the hot rolled strip. The average carbon content of the hot rolled strip is 0.07% at the edges and 0.05% at the Centre.

8. The best mechanical properties are obtained for the finished cold rolled and annealed direct strips which have been given 50-60% reduction in thickness during cold rolling. The strip, given a 60% reduction in thickness prior to annealing, has U.T.S. of about 250 MNm^{-2}, Y.S. of about 98 MNm^{-2} and about 38% elongation.

SUGGESTIONS FOR FURTHER INVESTIGATION

1. Coconut charcoal may be tried as a reductant for the block variant of direct strip process.
2. A detailed study may be done to understand the carburization mechanism of the porous sintered iron mass.
3. A careful scanning electron microscope examination may be done on a large number of cold rolled and annealed direct strip specimens to ascertain whether microcracks are formed around inclusions during cold rolling. Another, indirect method may also be tried for such study. The cold rolled strips, which have been given various amount of cold rolling, may be annealed at a relatively high temperature, say 1150°c, which would simultaneously bring about some sintering leading to removal of microcracks generated during the rolling. A comparision may then be made with mechanical properties of cold rolled direct strips annealed at lower temperature, say 700°c, as found in the present study. This comparision will give an indication whether the microcracks are formed around inclusions for higher amount of cold rolling or not.

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APPENDIX 1

THEORETICAL DENSITY OF MAGNETITE SUPERCONCENTRATE AND REDUCED SPONGE IRON

Constituents	Analysis of magnetite superconcentrate (W_1 %)	Analysis of reduced sponge iron on the basis of 100% chemical reduction (W_J %)	Density g/cc	Theoretical density	
				Magnetite superconcentrate	Reduced sponge iron
CaO	0.06	0.083	3.25		
MgO	0.175	0.242	3.58		
Al_2O_3	0.175	0.242	1.76		
SiO_2	0.075	0.104	2.64	5.3265 g/cc	7.8257 g/cc
Fe	71.750	99.330	7.86		
$FeO = \frac{72}{56}Fe^{++}$	30.600	-	5.70		
Fe_2O_3	68.500	-	5.18		

APPENDIX 2

THEORETICAL % WEIGHT LOSS FOR COMPLETE REDUCTION OF THE IRON ORE SUPERCONCENTRATE COMPACT

Weight percent of Fe = 71.7 - 71.8 (from chemical analysis)

Weight percent of Fe^{++} = 23.8 (")

Assuming the magnetite spinel to contain Fe^{++} and Fe^{+++} ions, the weight percent of Fe^{+++} = 47.95 %

During reduction only oxygen from magnetite is removed and the refractory oxides like CaO, SiO_2, Al_2O_3 and MgO remain unreduced.

% O_2 associated with Fe^{++} = $\frac{16}{55.8}$ x 23.8 = 6.82 %

% O_2 associated with Fe^{+++} = $\frac{48}{55.8 \times 2}$ x 47.95 = 20.624 %

∴ Total % O_2 to be removed for complete reduction = 27.45 %

Amount of binder solution used $\frac{\text{Ore}}{\text{Water}}$ = 20.

% Binder w.r.t. the ore = 1.

Let the weight of the ore be 'X' gms, then the amount of solid binder present in the compact = $\frac{x}{100}$ gms.

∴ Residue from the binder = $\frac{1.335}{100} \times \frac{x}{100}$ gms

∴ Weight of the fully reduced sponge iron block = Weight of iron ore superconcentrate (x gms) - Weight of O_2 associated with iron ions (0.2745 x gms) + weight of residue from binder (1.335×10^{-4} x gms).

∴ Theoretical weight loss percent for complete reduction of the iron ore superconcentrate compact = 27.44 %.

This value will be altered by the amount of clinkers sticking to the surfaces of the sponge iron block and the amount of carbon introduced into the sponge iron block during reduction.